

## PATENT ABSTRACTS OF JAPAN

(11)Publication number : 03-190996

(43)Date of publication of application : 20.08.1991

(51)Int.Cl.

C10M115/08  
C10M117/02  
C10M117/04  
C10M121/04  
// (C10M121/04  
C10M115:08 )  
(C10M121/04  
C10M115:04  
C10M113:08 )  
C10N 10:02  
C10N 10:04  
C10N 40:14  
C10N 50:10  
C10N 70:00

(21)Application number : 01-328208

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(22)Date of filing : 20.12.1989

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## (54) PRODUCTION OF GREASE HAVING EXCELLENT ACOUSTIC PROPERTY

(57)Abstract:

PURPOSE: To obtain a grease suitable for bearing having small diameter, etc., having noise-suppressing property by reacting a base oil dissolving or dispersing an isocyanate with a base oil dissolving or dispersing an amine in a specific condition.

CONSTITUTION: (A) A base oil dissolving or dispersing an isocyanate and (B) a base oil dissolving or dispersing an amine are respectively pressed at  $\leq 5\text{kg/cm}^2$  (preferably 25-200kgf/cm<sup>2</sup>) and reacted by impinging mixing, or respectively pressed at 0.5-25kgf/cm<sup>2</sup> (preferably 1-5kgf/cm<sup>2</sup>) and reacted with introducing to a stirring wing in rotating to afford the aimed grease.

## LEGAL STATUS

[Date of request for examination]

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's

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decision of rejection]

[Date of extinction of right]

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(57)

[WHAT IS CLAIMED IS:]

[Claim 1]

Production method of grease superior in acoustic properties; wherein; Isocyanate is dissolved or scattered base oil and amine are dissolved or scattered base oil is pressurized to (a) each, 25-200kg f/cm<sup>2</sup> in reaction vessel, it is introduced into the stirring feather which is rotating or it makes mix impact, and it is reacted or and it pressurizes to (b) each, to 25kg 0.5kgf/cm<sup>2</sup> f/cm<sup>2</sup>, and it is reacted.

[Claim 2]

Production method of grease superior in acoustic properties; wherein; Carboxylic acid or fat and fatty oil is dissolved or aqueous solution of hydroxide of hydroxide of base oil and the alkali metal which made disassemble or alkaline earth metal or decentralization fluid is pressurized to (a) each, 25-200kg f/cm<sup>2</sup> in reaction vessel, it is introduced into the stirring feather which is rotating or it makes mix impact, and it is reacted or and it pressurizes to (b) each, to 25kg 0.5kgf/cm<sup>2</sup> f/cm<sup>2</sup>, and it is reacted.

[Claim 3]

Claim 1 or production method of grease as claimed in 2; wherein; Mixing head is used as reaction vessel.

## [DETAILED DESCRIPTION OF THE INVENTION]

(a field of industrial application) the present invention relates to production method of grease having superior ability in the low noise characteristic which is claim character of grease employed by narrow path bearing. (prior art:) some proposal does method improving acoustic properties of grease conventionally. At first micro homogenization of grease is necessary for the first to improve acoustic properties of grease. In other words, That thickener component is left within grease, and it disperses uniformly is demanded. Subsequently, That foreign element in grease is removed is raised. As for the grease, it is classified roughly to the thing which does not assume that thickener component completely dissolves in base oil out of manufacturing process by temperature rising namely temperature. This is what the urea grease which uses isocyanate and amine for thickener charge stock can be said to. Among this urea grease, it is disclosed as for betterment method of only acoustic properties to completely dissolve by Japanese Patent Laid-Open No. 63-162790 bulletin by a this application person. Uniform decentralization of thickener component by completely dissolving was assured and these enabled removal of foreign element by mesh passage. On the other hand, It is thesis to disperse in uniformity in thickener constituent as for the only acoustic properties betterment method which is not completely dissolved out of manufacturing process, and validity of mil treatment in reaction operation termination time is disclosed by Japanese Patent Application No. 63-153654 specification by a this application person. In addition, Soap system grease employing hydroxide of hydroxide of carboxylic acid or fat and oil and alkali metal or alkaline earth metal in thickener charge stock is similar, and it is possible for uniformity decentralization of thickener constituent a thing to dissolve complete in and but removal of foreign element by mesh passage is possible, by way of example only, there are not guesses the betterment method as for uniformity decentralization of thickener constituent of lithium complex grease the thing which complete is not dissolved in. (only a problem to be solved by the invention) while doing, low noise called for by recent grease does not reach acoustic properties of the grease which complete does not dissolve strictness in within an increase, manufacturing process remarkably to be satisfied with even method described by Japanese Patent Application No. 63-153654 specification. When decentralization of thickener of grease is fixed at simultaneous in reaction time namely thickener micell formation, roller is massive, and this is thought about when there is limit in decentralization by the later me ring treatment. Therefore, It is method of acoustic properties betterment of the grease which a problem to be solved by the invention does not completely dissolve within manufacturing process, and it is in other words it is more advanced, and to disperse in uniformity within grease in thickener constituent.

(a means for solving problem) as a result that it should have been satisfied, and the scholars of present invention studied such a problem zealously, when decentralization of thickener component was done with generation of the thickener micell, sequence to be effective in extremely was reached. This is method to incorporate decentralization operation of thickener component in reaction operation, and in other words it is 1. in reaction operation of grease Isocyanate is dissolved or scattered base oil and amine are dissolved or scattered base oil is pressurized to (a) each, 25-200kg f/cm<sup>2</sup> in reaction vessel, it is production method of grease superior in acoustic properties including what it is introduced into the stirring feather which is rotating or it makes mix impact, and it is reacted or and it pressurizes to (b) each, to 25kg 0.5kgf/cm<sup>2</sup> f/cm<sup>2</sup>, and is reacted. 2. Carboxylic acid or fat and fatty oil is dissolved or aqueous solution of hydroxide of hydroxide of base oil and the alkali metal which made disassemble or alkaline earth metal or decentralization fluid is pressurized to (a) each, 25-200kg f/cm<sup>2</sup> in reaction vessel, production method of superior grease is related to in acoustic properties including what it is introduced into the stirring feather which is rotating or it makes mix impact, and it is reacted or and it pressurizes to (b) each, to 25kg 0.5kgf/cm<sup>2</sup> f/cm<sup>2</sup>, and is reacted. (a) compression is 25-200kg f/cm<sup>2</sup>, and preferably odor *te* is 1-5kg f/cm<sup>2</sup> in (b). As thus described it can be done at good beginning and end when it makes high pressure is pressurized in reaction vessel, and it collides, and mix reaction liquid, and responding and the thing that it makes low pressure introduce into the stirring feather which it pressurizes, and is rotating, and is responded use the high pressure pattern effervescence apparatus which is already put to practical use in polyurethane industry and low pressure pattern effervescence apparatus. Reaction vessel is preferable, and these respond in container as referred to as mixing head both. As for the high pressure type bubbling apparatus, impact mixes reaction liquid pressurized by high pressure in mixing head, it makes forming die discharge this reaction liquid in polyurethane industry with a thing to respond, reaction is traveled in pattern in a short time, polyurethane resin is molded by making do caking. In polyurethane industry, polyol and isocyanate are used in charge stock, but, it is maximal characteristic of that it can make extremely travel reaction / decentralization for effect because molecular order makes touch because both fluid pressurized by high pressure mixes impact. RIM (Reaction Injection Molding) is nominated for representation of this high pressure type bubbling apparatus. Low pressure type bubbling apparatus installs an agitation wing as referred to as an agitator in mixing head, this is turned in high speed, polyol and isocyanate are introduced here. Reaction / decentralization can make extremely travel this for effect by mechanical agitation power of this agitator. Next, It is not limited to polyurethane industry, and application of high pressure / low pressure bubbling apparatus is applied to Plastic Kogyo broadly, but, it is a present invention original thing as for application as against grease industry and is new. In addition, As for the present invention, a process after front of it is not limited in particular to relate to reaction operation when grease is produced. In other words, Hydroxide of isocyanate, amine to respond and aliphatic acid, density as opposed to base oil of fat and oil, temperature and alkali metal, density as opposed to aqua of hydroxide of alkaline earth metal, temperature can set optionally and the later operation namely heat, stirring, kneading, me ring apparatus can set optionally. In addition, Isocyanate is dissolved or scattered base oil and amine are dissolved or the present invention pressurizes scattered base oil to (a) each, 25-200kg f/cm<sup>2</sup> in reaction vessel in reaction operation when grease is produced, it makes mix impact, and it is reacted or (b) each, it is introduced into the stirring feather which it pressurizes, and is rotating, and responding to 25kg 0.5kgf/cm<sup>2</sup> f/cm<sup>2</sup> and carboxylic acid or fat and oil are dissolved or aqueous solution of hydroxide of hydroxide of scattered base oil and alkali metal or alkaline earth metal or decentralization fluid is pressurized to (a) each, 25-200kg f/cm<sup>2</sup> in reaction vessel, it is characterized by what it makes it makes mix impact, and it is reacted or (b) each, to 25kg 0.5kgf/cm<sup>2</sup> f/cm<sup>2</sup> introduce into the stirring feather which they pressurize, and is rotating, and is responded, if reaction / decentralization under condition to cut is a possible thing, even apparatus without designation of high pressure effervescence apparatus or low pressure effervescence apparatus is available for object of the invention to have realized. Even more particularly, This production method is very effective as against the grease which is not completely dissolved, but, it can be applied to for the thing which this reaction operation dissolves complete in, what in other words can be applied to all urea system and soap system grease, of course. In the present invention,

If base oil is an available thing as grease base oil, it can employ without mineral oil, otherwise of synthetic oil, restriction does not do particularly. By way of example only, The purification mineral oil which did paraffin series mineral oil, naphthenic mineral oil, hydrofining for mineral oil is given. In addition, It is silicone oil represented for synthetic oil by jikochirusebaketo, pentaerythritol ester, ester synthetic oil represented by dipentaerythritol ester, synthesis hydrocarbon oil represented by poly alpha olefin, ether system synthetic oil represented by alkyl diphenyl ether, dimethyl silicone. In addition, Isocyanate is 4,4 diphenyl-methane -  $\square$  diisocyanate (MDI), 4,4 tolylene diisocyanate (TDI), 3,3  $\square$  dimethyl diphenyl  $\square$  diisocyanate (TODI), and amine is alicyclic amine such as aniline, p - toluidine, p - chloroaniline, aromatic amine of naphthylamine, hexylamine, octyl amine, decyl amine, dodecylamine, hexadecyl amine, octadecylamine, aliphatic amine of eicosyl amine, cyclohexylamine. For carboxylic acid, it is polycarboxylic acid such as caproic acid, caprylic acid, capric acid, lauric acid, myristic acid, palmitic acid, stearic acid, hydroxy fatty acid of, 16- long chain fatty acid, 12 hydroxystearic acid, 12 hydroxy lauric acid hydroxy palmitic acid of behenic acid, sebacic acid, azelaic acid, oxalic acid, malonic acid, succinic acid, glutaric acid, adipic acid, pimelic acid, suberic acid, undecane diacid, dicarboxylic acid of dodecane diacid, other, dimer acid, trimer acid. In addition, It is the thing which did hydrogenation in beef tallow, animal fat and oil of lard, castor oil, rape seed oil, vegetable oil of coconut oil and these for fat and fatty oil or the thing which, even more particularly, was refined. In addition, Hydroxide of hydroxide of alkali metal and alkaline earth metal is lithium hydroxide, sodium hydroxide, potassium hydroxide, calcium hydroxide, barium hydroxide. In addition, To grease produced by this production method, additive such as oxidation inhibitor, antirust, oiliness agent, abrasion resistance, extreme pressure agent, solid lubrication agent can be doped if necessary. Next, The present invention is explained by drawing. An outline of high pressure type bubbling apparatus used with the present invention by figure 1 is shown. As is shown, it is from bent axis type axial piston pump 4, mixing head 3, hydraulic power package 5 as work reservoir 1 of isocyanate liquid, work tank 2 of amine fluid and cycle line, meter ring pump. According to the present invention, Reaction / dispersion of roller is done in mixing head. Isocyanate liquid, amine liquid is a thing when urea system grease is produced here isocyanate is dissolved in base oil or amine is dissolved in base oil or and amine fluid is scattered fluid about scattered fluid. The following describes a case of manufacture of urea system grease, but, it is each carboxylic acid or fat and oil is dissolved in base oil or hydroxide of scattered fluid, hydroxide of alkali metal or alkaline earth metal is dissolved in aqua or to teach work tank 1,2 scattered fluid on the occasion of soap system grease manufacturing. Before responding, it circulates. While it circulates, both aqua is pressurized in mixing head, but, circulation line is come back to without coming into contact by control piston. Within this circulation, a fine adjustment of discharge quantity of both aqua is done. It is drastic, and it is mixed impact in chamber of mixing head, and, on the occasion of reaction, the quantity fluid which control piston operates, and is pressurized is discharged as urea grease reactant. Low pressure type bubbling apparatus is same as the apparatus that high pressure mold foams as for the basic line. But, Hydraulic power package as shown in figure 1 completely has an agitation feather as referred to as an agitator in mixing head again, it rotates at speed. It is turned to this agitator on the occasion of reaction, and both aqua is jetted than branch. Compression of both aqua of this time is not high pressure like high pressure mold effervescence apparatus only in compression by pump, but, it is drastic, and is mixed by spin of an agitator, is discharged as urea grease reactant. Manufacturing process of grease with the use of this high pressure type bubbling apparatus is as follows. Base oil and isocyanate compound are added in work reservoir of isocyanate liquid, base oil and amine compound are doped in work tank of amine fluid. In doing so, it is warmed to 60-80 degrees Celsius depending on being essential each. Next, Blowout or the isocyanate liquid which became dispersion state, amine fluid are glued, it is done with  $N_2$  gas atmosphere. Next, It circulates, and line is warmed. Discharge quantity is decided so that it is in aequalis equivalent ratio in isocyanate liquid, amine fluid than batching orifice, impact blending namely reaction disperses in mixing head, and isocyanate fluid, amine fluid are discharged in voluntary delivery pressure. Grease spitted out is liquid in branch passage, and temperature before both phase reaction is desirable, and the temperature is 60-80 degrees Celsius, but, regurgitation rear hangs promptly hardened block-shaped solid or the temperature for several hours, and it continues rising to 130-140 degrees Celsius. When this reactant was received in container of storage drum, the

temperature of content held 100 degrees Celsius neighborhood later on 2nd. In addition, When grease was produced by means of low pressure type bubbling apparatus, it was done in similar for the case high pressure pattern effervescence apparatus. The condition of grease spitted out was a case using high pressure type bubbling apparatus and similar. Logically these phenomena are not yet established, but, is estimated like next. Preferably, in a case using high pressure type bubbling apparatus, both fluid pressurized to to  $200\text{kgf/cm}^2$  passes orifice more than  $5\text{kgf/cm}^2$ , and it is drastic, and it is mixed impact with impingement flow in mixing head. Both component of this time, isocyanate and amine, in molecular order, when is touched, it is conceivable, and is discharged by delivery. Reaction is not yet concluded with regurgitation event than contact time being short, it wants to be done, and it is liquid in condition in regurgitation. However, isocyanate and amine touch the thing which was popular with container in molecular order, even if there is not stirring, reaction travels, and temperature rise by exothermic reaction is continued. As thus described, As for making both phase come in contact with reaction time in molecular order, reactant namely thickener corpuscule is just to disperse in near form in molecular order. As for the hardness of grease, according to the morphology of the thickener grain, roller is massive, but, it is thought that thickener morphology in near form shows increase butterfly capacity of limit in this molecular order, the reason why it is in block-shaped solid can describe. In addition, Because, a case using low pressure type bubbling apparatus, contact in molecular order was enabled by high speed spin of an agitator, when effect same as a case using high pressure pattern effervescence apparatus appeared, it is conceivable. As is apparent from this real phenomenon side, isocyanate fluid and amine fluid are pressurized larger than each,  $5\text{kgf/cm}^2$  in carbuncle when high pressure type or low pressure pattern effervescence apparatus was applied to, when it makes or it makes mix impact, and it is reacted or each  $0.5\text{kgf/cm}^2$  -  $25\text{kgf/cm}^2$  introduce into the stirring feather which it pressurizes, and is rotating, and it is reacted, it can make isocyanate, both amine constituent touch in molecular order, the urea thickener corpuscule which, as a result, was generated gets possible to make there be in molecular order within grease in near form. In addition, In this way, as for the grease with the use of provided base grease, thickener component is uniform, and is dispersed within grease, as a result, it is thought that it can be with superior grease in acoustic properties. (embodiment and a comparative example) the present invention is explained by means of embodiment, a comparative example concretely. As for the embodiment, contents of a comparative example are shown in table 1,2, but, check method in here is as follows. (a) It was examined by optical microscope observation (magnification 100 times) (c) bearing sound check *anderonmeta* of (b) grease based on butterfly degree JIS K 2220 5.3. Check condition bearing 608 Thrust load  $2\text{kgf}$  Radial load  $150\text{gf}$  Number of revolutions 1800rpm Quantity of grease charge  $0.35\text{ml}$  Check time It is evaluated for two minutes It is shown with a mark (one hundred perfect score) than the number of the noise, result of *anderonreberu*. In addition, The embodiment, the base oil of a comparative example employed the following things. It is equal to or less than, and viscosity of alkyl diphenyl ether oil PA O ..... 40 degrees Celsius of 100cSt shows correction method of used grease in poly alpha *orenfin* oil of 100cSt and embodiment, a comparative example viscosity of ADE ..... 40 degrees Celsius. Be necessary in TDI5.53kg, ADE18.75Kg in work reservoir of example 1 isocyanate liquid, and work tank of amine fluid need in para toluidine 5.95kg, para chloroaniline 1.02kg, ADE18.75Kg, and equivalent adjusts discharge quantity in set pressure  $150\text{kgf/cm}^2$  of both tank after warming to 70-80 degrees Celsius in both fluid so that is responded, it was responded by high pressure pattern effervescence apparatus shown in figure 1. Because the reaction product became block-shaped solid, it was kneaded in usable hardness as grease, and urea grease of example 1 was got. The kneading apparatus used colloid mill made in *furima* company (300 clearance  $\mu\text{m}$  of rotor and suteta). As follows, Colloid mill made in *furima* company abbreviates to *furimamiru*. Urea grease of example 2 was got similarly except that *furimamiru* of example 2 example 1 was changed in three-high roll mill. Grease after reaction got in example 3 example 1 is raised in 1 degree Celsius /min, it holds at 175 degrees Celsius  $\square$  5 degrees Celsius for 30 minutes, it was kneaded in *furimamiru* after cooling (300 clearance  $\mu\text{m}$  of rotor and suteta), and urea grease of example 3 was got. Urea grease of embodiment 4 was got similarly except that *furimamiru* of four embodiment example 3 was changed in three-high roll mill. TDI4.77kg, ADE19.64kg are put in work reservoir of five embodiment isocyanate liquid, para toluidine 5.08kg, para chloroaniline 0.87kg, ADE19.64kg were put in work tank of amine fluid. As follows, It was

done same as example 1, and urea grease of embodiment 5 was got. TDI 3.94kg, ADE 20.55kg are put in work reservoir of six embodiment isocyanate liquid, para toluidine 4.24kg, para chloroaniline 0.72kg, ADE 20.55kg were put in work tank of amine fluid. As follows, It was done same as example 1, and urea grease of embodiment 6 was got. Urea grease of embodiment 7 was got similarly except that base oil of seven embodiment example 1 was changed in PA O. TDI 5.38kg, ADE 20.00kg are put in work reservoir of eight embodiment isocyanate liquid, para toluidine 4.62kg, ADE 20.00kg were put in work tank of amine fluid. As follows, It was done same as example 1, and urea grease of embodiment 8 was got. Work tank of isocyanate aqua of nine embodiment low pressure type bubbling apparatus needed in TDI 5.53kg, ADE 18.75Kg, and work tank of amine fluid needed in para toluidine 5.95kg, para chloroaniline 1.02kg, ADE 18.75Kg, and, after warming, equivalent adjusted discharge quantity so that it was responded, and 70-80 degrees Celsius were responded in both fluid. The delivery pressure of both aqua of this case was  $2\text{kgf/cm}^2$ . Because it was with block-shaped solid same as a case using the apparatus that high pressure type foamed as for the reaction product, it was kneaded in *furimamiru*. TDI (tolylene diisocyanate) 5.53kg, ADE 18.75kg were put in container of comparative example 1 the first, and it was warmed to 70-80 degrees Celsius. Para toluidine 5.95kg, para chloroaniline 1.02kg, ADE 18.75kg are taken in the second container, and it is warmed to 70-80 degrees Celsius, it was stirred as well as the first vessel. It is reaction method of case of urea grease production done to the public conventionally, and this is described as conventional method during following table 2. It was kneaded in *furimamiru* (300 clearance mu m of rotor and suteta), and, after reaction, urea grease of comparative example 1 was got. Grease after the reaction which went with two comparative example comparative examples 1 is raised in 1 degree Celsius /min, it holds at  $175 \pm 5$  degrees Celsius for 30 minutes, it was kneaded in *furimamiru* (300 clearance mu m of rotor and suteta) after cooling, and urea grease of comparative example 2 was got. Urea grease of comparative example 3 was got similarly except that *furimamiru* of three comparative example comparative examples 2 was changed in three-high roll mill. In blending of four comparative example comparative examples 1, *sutatekkumikisa* is used for reaction apparatus, a process of comparative example 2 was carried out, and urea grease of comparative example 4 was got. Base oil of five comparative example comparative examples 1 was changed into PA O, and a process of comparative example 2 was done, and urea grease of comparative example 5 was got.

表 1

	実施例 1	実施例 2	実施例 3	実施例 4	実施例 5	実施例 6	実施例 7	実施例 8	実施例 9
イソシアネート液 種類	TDI	TDI	TDI	TDI	TDI	TDI	TDI	MDI	TDI
重量 (kg)	5.53	5.53	5.53	5.53	4.77	3.94	5.53	5.53	5.53
基油 (kg)	18.75	18.75	18.75	18.75	19.65	20.55	18.75	20.00	18.75
アミン液 種類/重量 (kg)	パラトルイジン 5.95 パラクロロ アニリン 1.02	パラトルイジン 5.95 パラクロロ アニリン 1.02	パラトルイジン 5.95 パラクロロ アニリン 1.02	パラトルイジン 5.95 パラクロロ アニリン 1.02	パラトルイジン 5.08 パラクロロ アニリン 0.87	パラトルイジン 4.24 パラクロロ アニリン 0.72	パラトルイジン 5.95 パラクロロ アニリン 0.87	パラトルイジン 4.62	パラトルイジン 5.95 パラクロロ アニリン 1.02
基油 (kg)	18.75	18.75	18.75	18.75	19.64	20.55	20.55	20.00	18.75
基油の種類	ADE	ADE	ADE	ADE	ADE	ADE	PAO	ADE	ADE
反応装置	高圧型発泡装置								
反応後ミル	フリーマー	ロール	なし	なし	フリーマー	フリーマー	フリーマー	フリーマー	フリーマー
昇温の有無	無	無	有	有	無	無	無	無	無
昇温後ミル	—	—	フリーマー	ロール	—	—	—	—	—
ちょう度 CM/60N	246/251	233/245	233/235	230/231	233/235	324/328	355/358	364/369	275/281
光学顕微鏡写真 100倍	増ちょう剤粒子の分散は極めて良好であり、 $10\mu\text{m}$ 以下の粒子径で分散している。								
アンデロン 得点	81	82	92	94	88	89	71	72	80



表 2

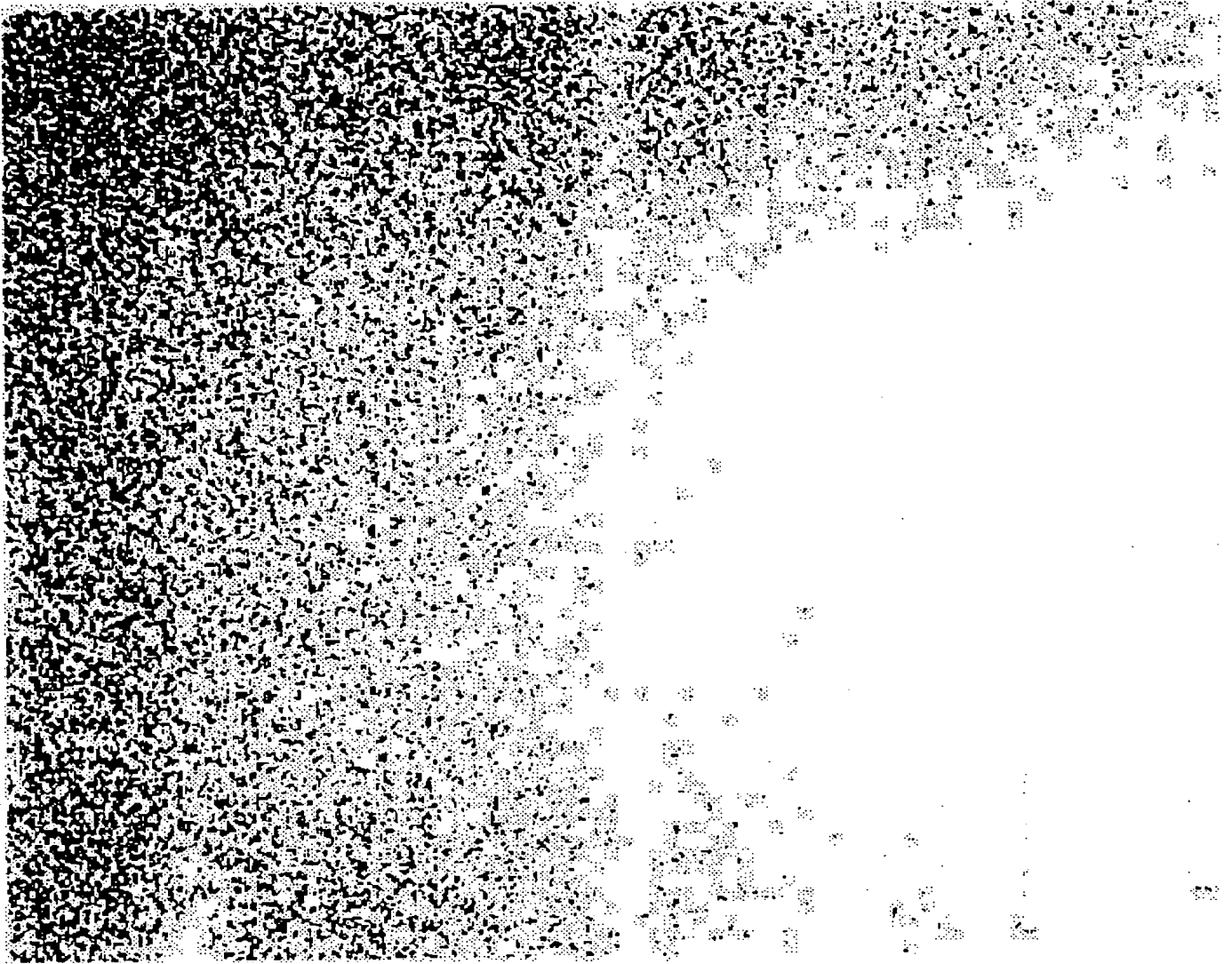
	比較例 1	比較例 2	比較例 3	比較例 4	比較例 5
イソシニアネート液 種類	T D I	T D I	T D I	T D I	T D I
重量 (Kg)	5.53	5.53	5.53	5.53	5.53
基油 (Kg)	18.75	18.75	18.75	18.75	18.75
ア ミ ソ 液 種類 / 重量 (Kg)	パラトルイジン 5.95 パラクロロ アニリン 1.02 18.75	パラトルイジン 5.95 パラクロロ アニリン 1.02 18.75	パラトルイジン 5.95 パラクロロ アニリン 1.02 18.75	パラトルイジン 5.95 パラクロロ アニリン 1.02 18.75	パラトルイジン 5.95 パラクロロ アニリン 1.02 18.75
基油 (Kg)	18.75	18.75	18.75	18.75	18.75
基油の種類	A D E	A D E	A D E	A D E	P A O
反応 方法	従来法	従来法	従来法	スタティックミキサー	従来法
反応後ミル	フリーマー	なし	なし	なし	なし
昇温の有無	無	有	有	有	有
昇温後ミル	—	フリーマー	ロール	フリーマー	フリーマー
ちょう度 0W/60W	261/261	281/285	272/274	273/275	293/295
光学顕微鏡写真 100倍	増ちょう剤粒子の大きさが数100 ~ 数10 $\mu$ m 程度で分散されている。				
アンデロン 得点	5	15	18	22	8

It was a score high, and, as a result of present embodiment, comparative example, acoustic properties improved more than 70 points in *anderson* examination markedly by introducing high pressure type bubbling apparatus or low pressure pattern effervescence apparatus into reaction apparatus. In embodiment, all mill aftertreatment is done after reaction of bubbling apparatus, but, this used to do block-shaped solid in the shape of grease. It is clear that improvement of these acoustic properties is effect of reaction / decentralization by effervescence apparatus rather than a thing by mill aftertreatment in comparison with a comparative example. In addition, When temperature rising operation is added after reaction / decentralization by bubbling apparatus, it is the few, but, that

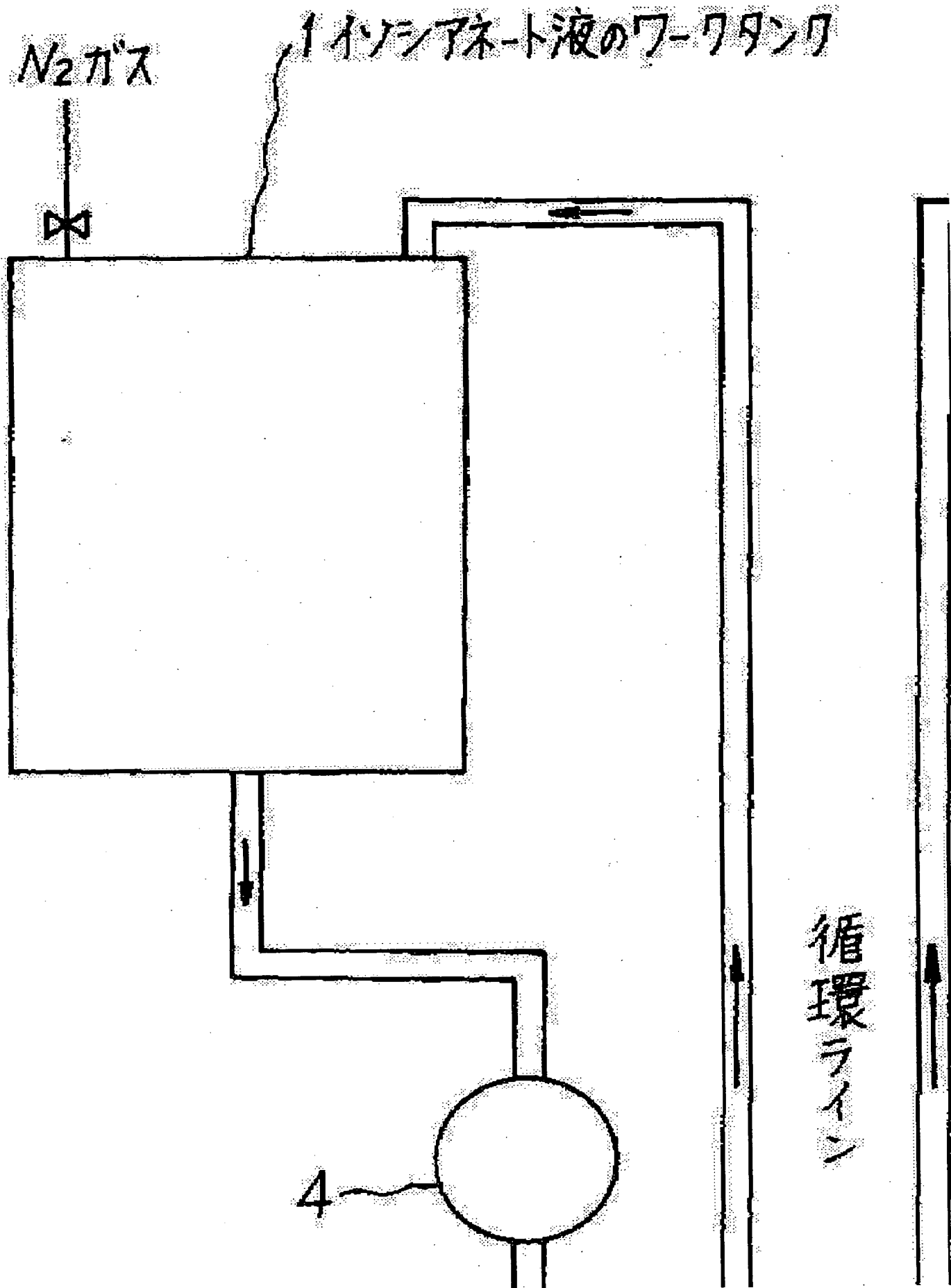
acoustic properties improved was recognized, but, this is effect on reaction / decentralization by effervescence apparatus, too. In addition, The effect that was homogeneous decentralization of thickener component mentioned a reason of improvement of these acoustic properties above, but, light microscope photography of urea grease of example 1 and comparative example 1 (100 times) is shown in figure 3 that figure 2 invites corpuscule configuration of urea grease as a representative example. At a glance, as can be appreciated, grease of example 1 is homogeneous, and there is not ten  $\mu\text{m}$  which seem to be seen in comparative example 1 in corpuscule older than. Even other embodiment, a comparative example are the same, and this disperses in uniformity in thickener constituent in a thing with the use of bubbling apparatus, that acoustic properties were improved is recognized. (an effect of the invention) 1. isocyanate is dissolved or scattered base oil and amine are dissolved or scattered base oil is pressurized larger than (a) each,  $5\text{kgf/cm}^2$  in reaction vessel, 2. thing carboxylic acid it is introduced into the stirring feather which it pressurizes, and is rotating, and to react or fat and oil is dissolved in it makes mix impact, and it is reacted or (b) each,  $0.5\text{kgf/cm}^2$  -  $25\text{kgf/cm}^2$  or aqueous solution of hydroxide of hydroxide of base oil and the alkali metal which made disassemble or alkaline earth metal or decentralization fluid is pressurized larger than (a) each,  $5\text{kgf/cm}^2$  in reaction vessel, it is compared with grease provided by means of conventional production method with the present invention including what is reacted or it makes mix impact, and it is reacted or and it is introduced into (b) each,  $0.5\text{kgf/cm}^2$  - the stirring feather which it pressurizes to  $25\text{kgf/cm}^2$ , and is rotating, uniformity decentralization of thickener constituent is assured, the grease which could improve acoustic properties of bearing gets possible to be got.

#### [BRIEF DESCRIPTION OF DRAWINGS]

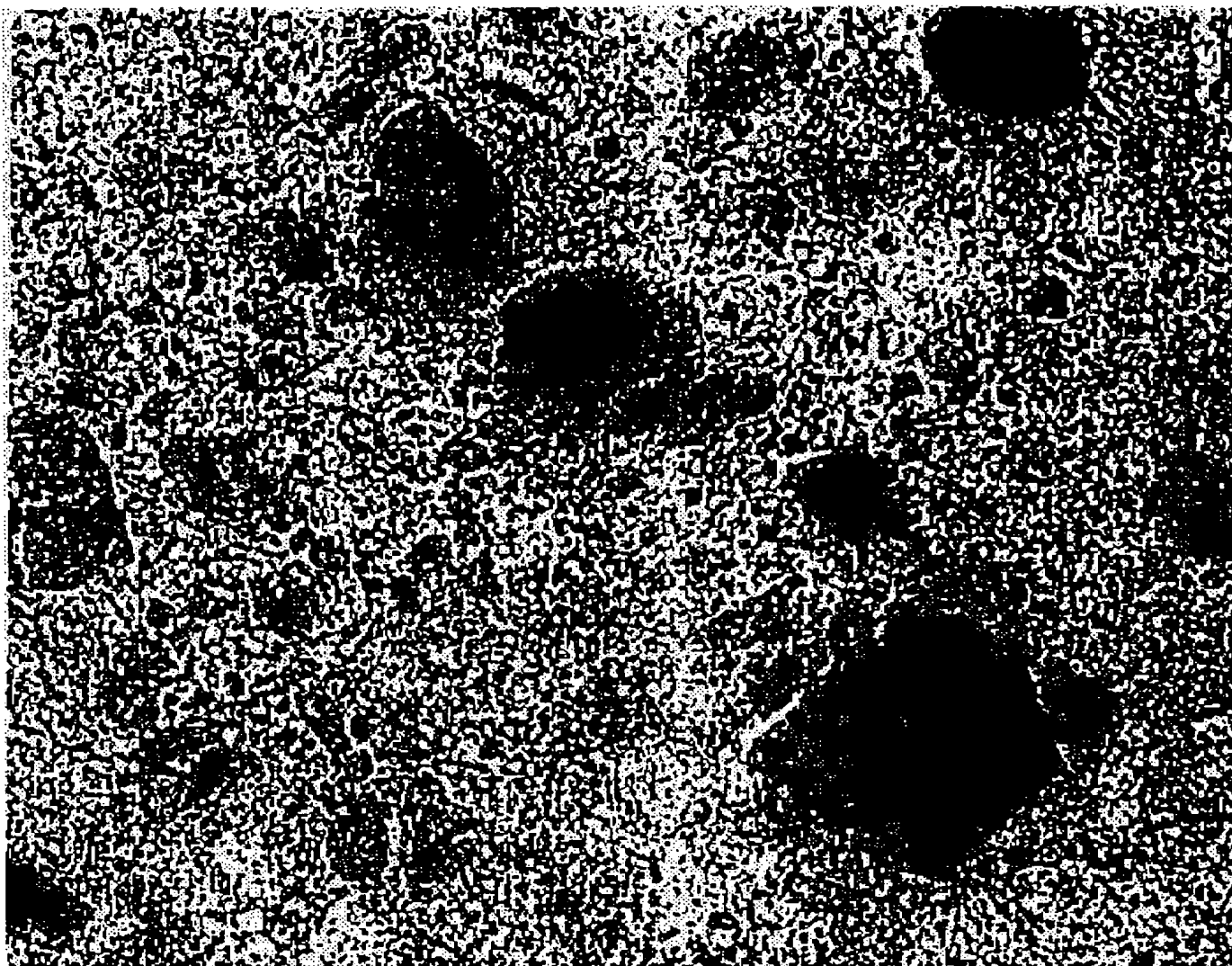
Light microscope photography, figure 3 that figure 1 shows corpuscule configuration of urea grease of example 1 in plot plan, figure 2 to show an outline of high pressure type bubbling apparatus used for method of the present invention are light microscope photography to show corpuscule configuration of urea grease of comparative example 1 in. Three work tank ..... mixing head of two work reservoir ..... amine liquid of one ..... isocyanate liquid, four five ..... pump ..... hydraulic power package.



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